EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title: Uncovering the mesoscale morphological evolution of high-performance semi-aromatic thermoplastic copolyesters**	**Experiment number**: A26-2-960
Beamline: BM-26	**Date of experiment**: from: 18 July 2023 to: 22 July 2023	**Date of report**: 15 August 2023
Shifts: 12	**Local contact(s)**: Martin Rosenthal (martin.rosenthal@esrf.fr)	*Received at ESRF:*

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Report:

This experiment involved performing simultaneous SAXS and WAXS measurements on a variety of thermoplastic copolyester block copolymers. The primary aim was to evaluate the evolution of morphologies in these segmented copolymers during different phase transformations. The primary architecture of the copolymers is illustrated in Figure 1, in which there are alternating hard, semi-crystalline segments and soft, low T_g segments in a multi-block type architecture.

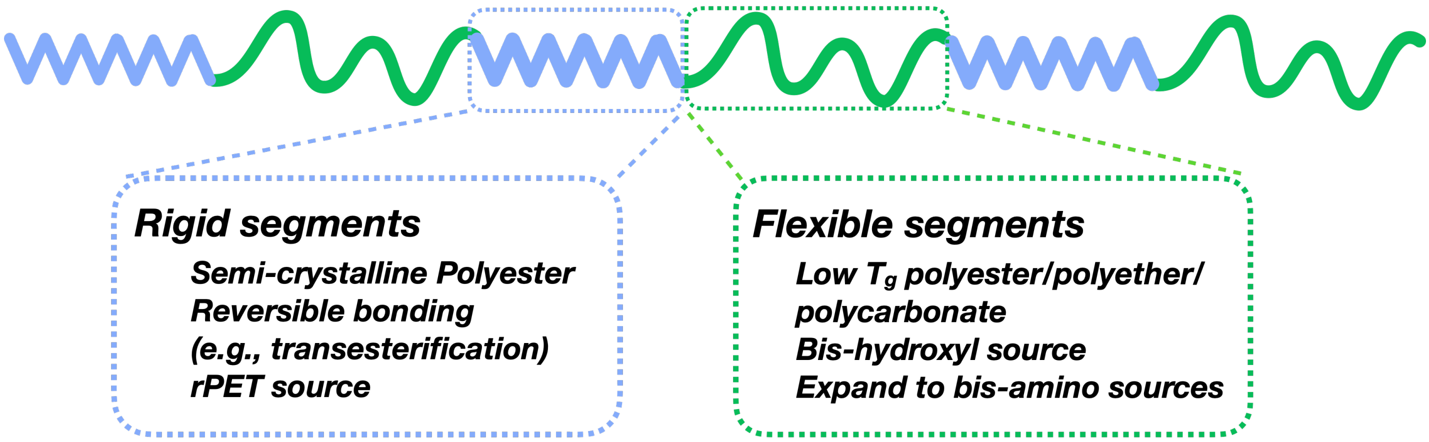

Figure 1. Cartoon representation of the multiblock copolymers investigated in this experiment.

We had a variety of different copolymers, differing in both the molecular identity of the hard block (PET vs. PBT) and the composition (i.e., weight/mass percent of the respective blocks). Using this diverse sample set, we explored various different morphological/phase transitions. Initially, we investigated the temperature dependence of phases, heating and cooling the samples while capturing SAXS/WAXS frames. From this we are able to identify both phase sizes (i.e., crystalline domain size) and also approximate melting temperatures coinciding with the morphological transitions. We are looking for the different transitions in copolymers with different compositions, and also different backbone type. Figure 2 shows examples of data obtained from these heating/cooling experiments, where a distinct difference can be seen in the transition temperature from copolymers containing either PET or PBT hard segments.

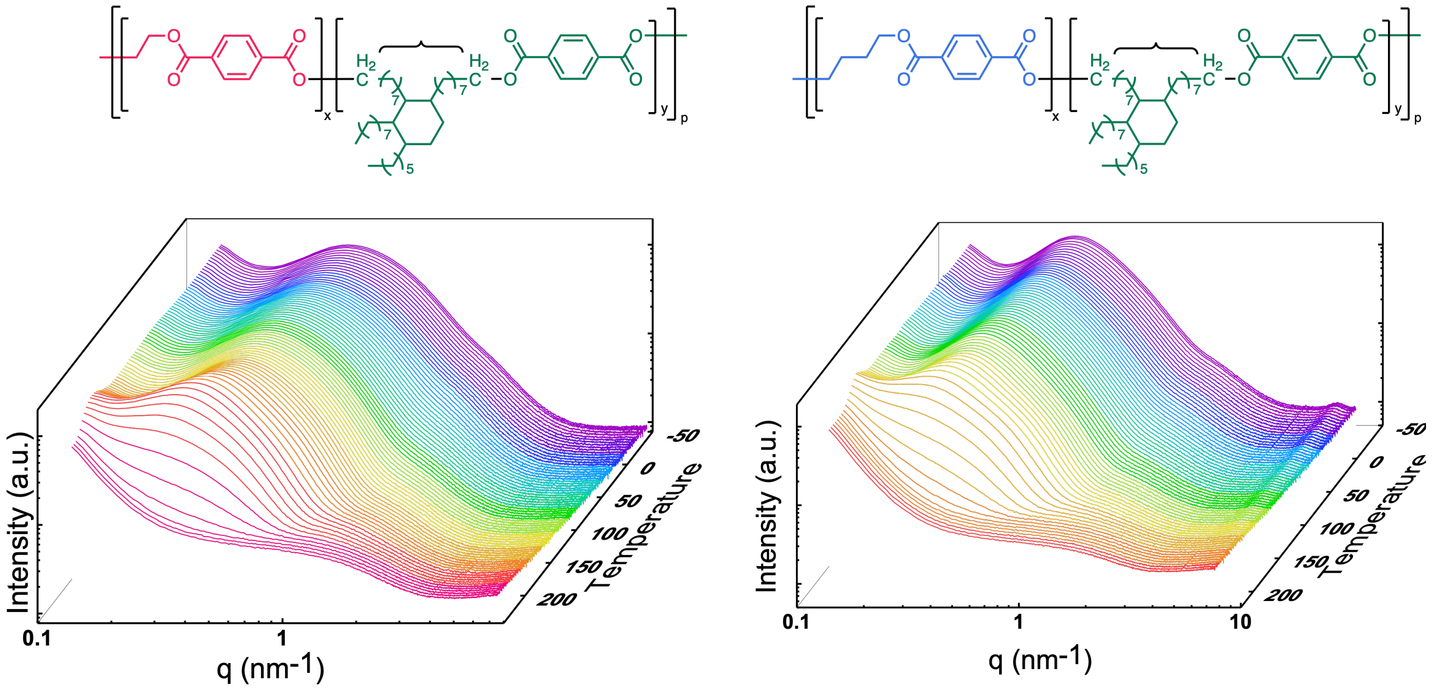

Figure 2. Representative heating in situ SAXS measurements for copolymers containing PET hard segments (left) or PBT segments (right).

In addition to the heating/cooling, we also performed a series of SAXS/WAXS measurements on the same sample set during mechanical/tensile deformation. The in situ deformation experiments revealed the changes in morphologies during deformation, an imporant indicator of performance in these engineering materials. Crystalline lamellae befome deformed on a mesoscale and rearrange or reorient themselves upon deformation, which has a dramatic influence on the mechanical performance. This relationship can now be revealed from the data captured during this experiment. We plan on publishing this data in at least two separate publications, which we are working on now. We plan follow up experiments based on the valuable data captured, using different copolymers.